इंटरनेट मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”
Mazdoor Kisan Shakti Sangathan
“The Right to Information, The Right to Live”

“पुराने को छोड नये के तरफ”
Jawaharlal Nehru
“Step Out From the Old to the New”

IS 538 (2000): Phenol (Carbolic Acid) [PCD 3: Petroleum, Lubricants and their Related Products]

“ज्ञान से एक नये भारत का निर्माण”
Satyanarayan Gangaram Pitroda
“Invent a New India Using Knowledge”

Act Number 22 of 2005
The Indian Parliament
RIGHT TO INFORMATION

Made Available By
Public.Resource.Org

“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”
Bhartṛhari—Nītiśatakam
“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE

BRUTUM FULMEN

IGNORANTIA JURIS NON EXCUSAT

PROTECTED BY COPYRIGHT

भारतीय मानक

फिनॉल (कारबोलिक एसिड) — विशिष्टि

(दूसरा पुनरीक्षण)

Indian Standard

PHENOL (CARBOLIC ACID) — SPECIFICATION

(Second Revision)

ICS 71.080.90

© BIS 2000

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

November 2000

Price Group 5

Aromatic Hydrocarbons and Related Products Sectional Committee, PCD 8

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by Aromatic Hydrocarbons and Related Products Sectional Committee had been approved by the Petroleum Coal and Related Products Division Council.

Phenol is obtained from coal tar, in the fraction distilling between 170 °C and 230 °C. The reaction of sodium hydroxide is applied in the recovery, followed by treatment with carbon dioxide and distillation of the separated phenol. It is also obtained from benzene sulphonic acid ($C_6H_5SO_3H$) by heating with sodium hydroxide or calcium hydroxide; or from chlorobenzene by heating under pressure with sodium hydroxide in the presence of diphenyl oxide.

Phenol is used as an antiseptic, germicide and disinfectant. It is also used in the manufacture of certain dyes and picric acid.

This standard was first issued in 1955 and revised in 1968. In the revised version requirement for boiling point was omitted and use of a dehydrated sample in the determination of crystallization point was stipulated. However, the committee responsible for preparation of this standard decided to revise it again in accordance with the development in and growing demand by the petrochemical industry. Cognizance has been taken of the developments in overseas specifications *vis-a-vis* requirements in Indian conditions. Thus the present standard has been updated in accordance with the need of the industry and in the light of the past experience gained.

Therefore in the present version (Second Revision) of the standard two grades of the material have been introduced on the basis of assay and crystallization point. The grades are also meant for specific use like pharmaceuticals and for general industrial purposes. The GC analysis has been included as an alternative method for determination of purity.

In the preparation of this standard, considerable assistance has been derived from the following publications:

ISO 1897 : 1977	Phenol, *o*-cresol, *m*-cresol, *p*-cresol, cresylic acid and xylenols for industrial use — Methods of test
ISO 1897-1 : 1977	Part 1: General
ISO 1897-2 : 1977	Part 2: Determination of water-Dean and Stark method
ISO 1897-3 : 1977	Part 3: Determination of neutral oils and pyridine bases
ISO 1897-4 : 1977	Part 4: Visual test for impurities insoluble in sodium hydroxide solution (Excluding cresylic acid and xylenols)
ISO 1897-5 : 1977	Part 5: Visual test for impurities insoluble in water (Phenol only)
ISO 1897-10 : 1982	Part 10: Determination of dry residue after evaporation on a water bath (Excluding cresylic acid and xylenols)
ISO 1897-11 : 1983	Part 11: Determination of crystallizing point (Excluding cresylic acid and xylenols)
ASTM 4961 : 1993	Gas chromatographic analysis of major organic impurities in phenol produced by the cumene process
BS 523 : 1964	Specification for phenol (withdrawn)

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
PHENOL (CARBOLIC ACID) — SPECIFICATION
(Second Revision)

1 SCOPE

This standard (Second Revision) prescribes the requirements and the methods of sampling and test for phenol (carbolic acid).

2 NORMATIVE REFERENCES

The following standards contain provisions which through, reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
265 : 1993	Specification for hydrochloric acid (*fourth revision*)
1070 : 1992	Reagent grade water (*third revision)*
1260 (Part 1) : 1973	Pictorial marking for handling and labeling of goods : Part 1 Dangerous goods (*first revision*)
2362 : 1973	Determination of water by the Karl Fischer method (*first revision*)
4905 : 1968	Methods for random sampling
5296 : 1995	Chloroform, pure and technical (*second revision*)
1448 [P:40] : 1987	Methods of test for petroleum and its products [P:40] water by distillation (*first revision*)

3 GRADES

The material shall be of two grades, namely:

a) Grade A; and

b) Grade B.

4 REQUIREMENTS

4.1 Description

The material shall be colourless to light pink liquid and free from suspended matter at 41 °C. Phenol may also be supplied as hydrated phenol containing a specified quantity of water (4 to 10 percent by mass) as agreed to between the purchaser and the supplier. In such cases the phenol content of the hydrated phenol shall be determined by the Bromination method and water content by the method of IS 1448 [P : 40] : 1967 using 2 or 10 ml receiver depending on water content and toluene as carrier liquid.

4.2 Solubility in Water

When 10 g of the material is mixed with 120 ml of distilled water, the solution shall be complete and clear, and shall remain so after standing for 30 minutes at 27 °C.

4.3 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in Annex A to D.

Table 1 Requirements for Phenol (Carbolic Acid)

Sl No.	Characteristic	Requirement		Method of Test, Ref to Annex
		Grade A	Grade B	
(1)	(2)	(3)	(4)	(5)
i)	Crystallization point, °C, *Min*	40.3	39.5	A
ii)	Moisture, percent by mass, *Max*	0.1	0.1	B
iii)	Residue on evaporation by mass, *Max*	0.05	0.05	C
iv)	Assay, percent by mass, *Min*	99.5	98.5	D

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in amber coloured or dark blue glass bottles fitted with ground glass stopper or as agreed to between the purchaser and the supplier.

5.2 Marking

5.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name and grade of the material;

b) Lot or batch number and date of manufacture;

c) Tare, gross and net mass; and

d) Indication of the source of manufacture.

5.2.2 Each container shall also be marked with Fig. 11 of IS 1260 (Part 1) along with the following information printed in the space provided:

POISON
'PROTECT FROM LIGHT AND MOISTURE'

5.2.3 *BIS Certification Marking*

The container may also be marked with the BIS Certification Mark.

5.2.3.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Annex E.

7 TEST METHODS

7.1 Tests shall be conducted as prescribed in Annex A to D.

7.2 Quality of Reagents

Unless otherwise specified, pure chemicals and distilled water (*see* IS 1070) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF CRYSTALLIZATION POINT

A-1 APPARATUS

A-1.1 Crystallization-Point Apparatus

The apparatus is illustrated in Fig. 1. A glass tube 150 × 25-mm nominal size is placed inside a 175 × 38-mm test tube. The latter tube is flanged so that it may be supported centrally by a metal cover plate, in a 1 000-ml tall-form beaker filled with water to within 20 mm of the top. The wider tube is weighed with lead shots or similar material and the inner tube is closed by means of a cork which carries a glass stirrer and through its centre a standard thermometer. The stirrer has a loop of outside diameter of about 18 mm, surround the thermometer. The thermometer is so fixed in the cork that the bottom of the bulb is about 15 mm from the bottom of the inner tube. A thermometer for the water-bath passes through a hole in the cover plate and is held by a rubber ring.

A-1.2 Thermometer

Conforming to the following essential requirements:

Range	15 to 45 °C
Graduation	0.1 °C
Longer lines, at each	0.5 and 1 °C
Fully figured, at each	2 °C
Overall length, *Max*	430 mm
Length of main scale, *Min*	260 mm
Error, *Max*	+ 0.4 °C

NOTES

1 Any thermometer of similar requirements and accuracy may be used.

2 The thermometer shall bear a certification from the National Physical Laboratory, New Delhi or any other institution authorized by the Government of India to issue such a certificate.

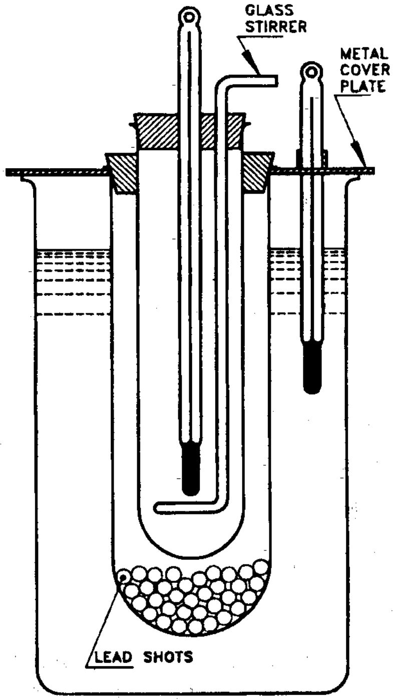

FIG. 1 APPARATUS FOR THE DETERMINATION OF CRYSTALLIZING POINT

A-1.3 Water-Bath

A-1.4 Measuring Cylinders

50 ml and 10 ml capacities.

A-2 PROCEDURE

A-2.1 Determination of Crystallization Point

Remove the inner tube from the crystallization point apparatus and directly pour about 20 ml of the sample (in liquid form) into it. Insert the cork carrying the thermometer and stirrer. If the sample has commenced to crystallize, heat gently until it becomes liquid and then cool rapidly to determine the approximate crystallizing point. Warm the tube in the water-bath at about 5 °C above this point, so that the crystals melt, except for a trace necessary for seeding. Replace the inner tube in its jacket with the water in the apparatus maintained at a temperature between 6 and 8 °C below the expected crystallizing point. Stir the sample gently and continuously and record thermometer readings at 30 s intervals. The crystallizing point corresponds to the highest of the first five consecutive readings during which the temperature remains constant within 0.1 °C.

If supercooling occurs, as shown by a rise in temperature, observe the constant temperature after the rise. A temperature rise of 1 °C is the maximum allowable. If five consecutive readings within 0.1 °C are not obtained, record six readings commencing with the point at which the maximum temperature is first attained. Plot the complete cooling curve of temperature against time and draw a straight line to lie evenly between the first and second and between the fifth and sixth points mentioned above. Extend this line to meet the section of the cooling curve before the temperature rise. Report the temperature corresponding to the point of intersection as the crystallizing point.

ANNEX B

[*Table* 1, *Sl No.*(ii)]

DETERMINATION OF MOISTURE

B-1 PROCEDURE

Determine the moisture content in phenol by Karl-Fischer method as prescribed in IS 2362.

ANNEX C

[(*Table* 1, *Sl No.* (iii)]

DETERMINATION OF RESIDUE ON EVAPORATION

C-1 APPARATUS

C-1.1 U-Tube- Stoppered

With limbs about 125 × 15 mm. Each limb is provided with a side tube as shown in Fig. 2. The stoppers shall not be greased.

C-1.2 Air Bath

A copper air bath about 150 ×150 ×150 mm.

C-1.3 Thermometer

Conforming to the following essential requirements:

Range	50 to 210 °C
Graduation	0.5 °C
Longer lines, at each	1 and 5 °C
Fully figured, at each	10 °C
Overall length, *Max*	430 mm
Length of main scale, *Min*	240 mm
Error, *Max*	+0.8 °C

NOTE — The thermometer shall bear a certificate from the National Physical Laboratory, New Delhi or any other institution authorized by the Government of India to issue such a certificate.

C-1.4 Calcium Chloride Tube

C-1.5 Conical Safety Flask

C-1.6 Suction Pump

C-2 PROCEDURE

C-2.1 Assemble the apparatus as shown in Fig. 2 without the U-tube. Heat the air bath to 150 ± 5 °C, and maintain it at this temperature. Connect the

U-tube and draw a gentle stream of air through it for approximately half an-hour. Remove the U-tube, connect it by a side arm to the calcium chloride tube and close the other side arm to the atmosphere. When cool, remove the calcium chloride tube and weigh the U-tube to within 0.000 1 g with both stoppers closed.

C-2.2 Weigh accurately about 10 g of the sample into the U-tube, at the lowest temperature for complete liquidity. Reconnect the U-tube to the train and make sure that the bottom of the thermometer bulb is level with the bottom of the U-tube.

C-2.3 Draw a gentle stream of air through the apparatus and maintain the air-bath at 150 ± 5 °C until evaporation is complete. Disconnect the U-tube, fit the calcium chloride tube to one side arm and close the other to the atmosphere. When the tube is cool, weigh it as before. Repeat the heating, cooling and weighing until a constant mass is obtained.

C-3 CALCULATION

$$\text{Residue on evaporation, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue left, and

M_2 = mass in g of the sample taken for the test.

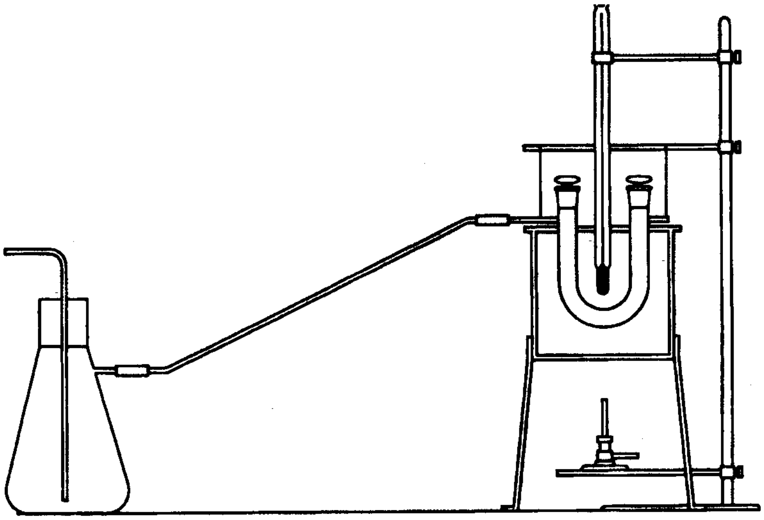

FIG. 2 APPARATUS FOR THE DETERMINATION OF RESIDUE ON EVAPORATION

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF ASSAY

D-1 GENERAL

Two methods have been specified for determination of assay of phenol Method A is bromide-bromate volumetric method while Method B is the gas chromatographic method. In case of dispute method A shall be the referee method.

D-2 BROMIDE-BROMATE SOLUTION METHOD (METHOD A)

D-2.1 Outline of the Method

The method is based on the reaction between phenol and bromine from standard bromide-bromate solution. Other phenols and unsaturated compounds react with bromine and if these are present they will invalidate the results.

D-2.2 Reagents

D-2.2.1 *Chloroform* (*see* IS 5296).

D-2.2.2 *Concentrated Hydrochloric acid* (*see* IS 265).

D-2.2.3 *Potassium Iodide Solution 20 percent* (m/v)

Dissolve 20 g of potassium iodide in 100 ml of water.

D-2.2.4 *Standard Bromide-Bromate Solution*

0.1 N

Dissolve 10.2 g of potassium bromide and 2.784 g of potassium bromate in water and dilute to 1 000 ml in a volumetric flask.

D-2.2.5 *Standard Sodium Thiosulphate Solution*

0.1 N

Dissolve 25 g of sodium thiosulphate in water and dilute to 1 000 ml. It is essential to find out the factor of the sodium thiosulphate solution immediately before use, as follows:

Pipette 25 ml of the bromide-bromate solution into the iodine flask, add 5 ml of the potassium iodide solution, followed by 5 ml of hydrochloric acid. Stopper immediately and shake thoroughly. Titrate the liberated iodine with the sodium thiosulphate solution until the contents of the flask become only faintly yellow; add a few drops of starch solution and continue the titration until the last traces of the blue colour have disappeared.

The factor 'f' of the sodium thiosulphate solution is 25/v, where v is the volume (ml) of sodium thiosulphate solution used.

D-2.2.6 *Starch Solution*

Freshly prepared.

D-2.3 Apparatus

D-2.3.1 *Iodine Flask*

500-ml capacity.

D-2.3.2 *Measuring Cylinders*

One of 100 ml and three of 25 ml capacities.

D-2.3.3 *Pipettes*

50 ml and 25 ml capacities.

D-2.3.4 *Burette*

50-ml capacity

D-2.4 Procedure

Weigh accurately about 2.5 g of the sample and dissolve it in water; dilute to 1 000 ml with water in a volumetric flask. Pipette 25 ml of this solution into the iodine flask and add 50 ml of the bromide-bromate solution, followed by 5 ml of hydrochloric acid. Stopper immediately and seal by running a little of the potassium iodide solution into the annular space between the stopper and the funnel-shaped neck of the flask. Mix the contents of the flask by occasional swirling during half an hour and then leave to stand for a further period of 15 minutes. Gradually add 15 ml of potassium iodide solution to the contents of the flask by pouring the solution into the annular space on the neck and gently easing the stopper. Shake the flask thoroughly and titrate the contents with the standard sodium thiosulphate solution until only a faint yellow colour remains. Add 10 ml of chloroform to dissolve the bulky precipitate of bromophenol which, otherwise, is liable to absorb iodine, and follow by a few drops of starch solution. Continue titrating and shaking the contents of the flask until the last traces of the blue colour have disappeared. Record the volume of the sodium thiosulphate solution used.

D-2.5 Calculation

Each ml of 0.1 N bromide-bromate solution consumed is equivalent to 0.001 568 g of phenol.

$$\text{Assay, percent by mass} = \frac{6.272\,(50 - fv)}{M}$$

where

f = factor for the standard sodium thiosulphate solution (*see* **D-2.2.5**);

v = volume, in ml, of the standard sodium thiosulphate solution used; and

M = Mass, in g, of the sample taken for the test.

D-3 GAS CHROMATOGRAPHIC METHOD (METHOD B)

D-3.1 General

This method determines the concentration of major impurities such as mesityl oxide, cumene, α-methylstyrene, 2-methylbenzofuran, acetophenone, and dimethylbenzyl alcohol.

D-3.2 Outline of the Method

A known amount of internal standard is added to the sample. A portion of the sample is analyzed by gas chromatography. The concentration of each impurity is calculated relative to the known amount of internal standard that is added. It is useful for individual impurities in the range 10 to 100 mg/kg.

D-3.3 Apparatus

D-3.3.1 *Chromatograph*

Equipped with an on-column injector and flame ionization detector.

D-3.3.2 *Recorder*

With a full scale of 1 mV and a response time of 1 s or less.

D-3.3.3 *Integrating Device*

Any device capable of integrating chromatograph peak areas with a repeatablility of ±1 percent relative.

D-3.3.4 *Chromatographic Column*

Glass (2-mm inside diameter) or Nickel 200 (5.3 mm inside diameter by 3 m length). The column may be purchased or prepared as described in **D-3.5**.

NOTE — *CAUTION* — Other nickel tubing may not be satisfactory because of the catalytic decomposition of compounds of interest that may occur in tubing other than Nickel 200.

D-3.4 Reagents and Materials

D-3.4.1 *Solid Support 100/120 Mesh*

Acid washed, dimethylchlorosilane (DMCS) treated diatomaceous earth.

D-3.4.2 *Stationary Phase*

A combination of 8 weight percent acidified polyethylene glycol plus 4 weight percent versamid 900 (*see* Note). A suitable solvent is 75 volume percent methylene chloride plus 25 volume percent toluene.

NOTE — Versamid 900 is a trademark of General Mills Inc, is available from any chromatographic supplier.

D-3.4.3 *Internal Standard*

Tetradecane.

D-3.4.4 *Pure Components for Calibration*

Mesityl oxide, cumene, α-mesitylstyrene, 2-methylbenzofuran, acetophenone, and dimethylbenzyl alcohol. The purity of each component should exceed 99 weight percent.

D-3.4.5 *Phenol*

High purity phenol.

D-3.4.6 *Carrier Gas*

Helium or nitrogen.

D-3.5 Column Preparation

D-3.5.1 Either glass or nickel columns may be used. Glass columns are preferred because of their superior inertness. Nickel columns are preferred because of ease of handling. If nickel tubing is used, it must be cleaned with glacial acetic acid, followed by water rinsing.

D-3.5.1.1 Prepare the column packing as 12 weight percent of the stationary mixed phase to 88 weight percent of the solid support. Dissolve the stationary phase in sufficient solvent so that the solution will cover the solid support completely in a flat, shallow, glass or porcelain vessel. Place the vessel in a ventilating hood and evaporate the solvent to a constant weight, with occasional gentle stirring. Add the free-flowing packing thus obtained to the column with the aid of a small funnel, and apply vacuum to draw the packing into place. Either tap or vibrate the column to ensure even distribution of the packing in the column. Place a glass-wool plug at the outlet end of the column before adding the packing, and one at the inlet end after the column in full. Do not pack the packing too tightly; evenly distribute it with no voids.

D-3.5.2 *Column Condition*

Install the column into the chromatograph and precondition it at the elevated temperature described in **D-3.5.2.1** through **D-3.5.2.4**. Do not connect the column to the detector block until **D-3.5.2.2** has been completed.

D-3.5.2.1 Start the flow of helium or nitrogen carrier gas and allow the column to age for 30 min with no heat.

D-3.5.2.2 Increase the column temperature to 230 °C at the rate of 2 °C/min and hold that temperature for at least 24 h. Connect the column to the detector block after this step been completed.

D-3.5.2.3 If a nickel column is used, reduce the temperature to 130 °C and make fifteen 1-μl injections of phenol.

D-3.5.2.4 Establish the conditions shown in Table 2 and make repetitive phenol injections until two or more injections exhibit the same peak configuration.

D-3.6 Preparation and Calibration of Standards

D-3.6.1 Use high purity phenol in preparing the calibration standards. Determine the residual impurities in the phenol by the procedure outlined in **D-3.7**.

D-3.6.1.1 *Sample preparation*

The sample must be handled in a molten state at 50 °C. Higher temperatures will degrade the sample.

D-3.6.2 Prepare a calibration mixture of phenol containing mesityl oxide (MO), cumene (CU), α-methylstyrene (AMS) 2-methylbenzofuran (2MBF), acetophenone (AP), and dimethylbenzyl alcohol (DMBA). All the above impurity levels should be near the anticipated levels in the sample. If any residual impurity elutes with a known impurity, deduct the residual area obtained in **D-3.6.1** (adjusted to sample size) from the area of the impurity.

D-3.6.3 *Standardization Procedure*

Determine the relative response factors (RRF) of each impurity by adding known quantities of impurities and internal standard. Prepare standards with impurity levels that bracket the dynamic range of interest. Use the procedure in **D-3.7** for standardization. Determine the relative response factor, F, as follows;

$$F = [\, W_c \times A_I / (A_c - A_b) \,] \times W_I$$

where

W_c = weight of impurity, g,

W_I = weight of internal standard, g,

A_I = peak area of internal standard,

A_c = peak area of impurity in calibration blend, and

A_b = peak area of impurity in phenol base stock.

Set the system sensitivity so that all impurity peaks are recorded at adequate levels for data acquisition. Normally, the minimum peak height will be twice that of the baseline noise.

Table 2 Typical Conditions for Chromatographic Separation

(*Clause* D-3.5.2.4)

Column temperature °C	180 isothermal
Injector temperature °C	180
Detector temperature °C	250
Carrier gas	nitrogen or helium
Gas flow rate, cm^3/min	22
Hydrogen flow rate, cm^3/min	30
Air flow rate, cm^3/min	320
Specimen size, µl	1

NOTE — Phenol produced by processes other than the cumene process may have impurities that elute with tetradecane. Before using this test method, investigate this by analyzing the phenol without the internal standard. If interference exists, use other internal standards such as durene or sec-butyl alcohol.

D-3.6.4 *See* Table 3 for typical response factors and retention times.

D-3.7 Procedure

D-3.7.1 *See* Table 2 for chromatographic conditions.

D-3.7.2 Add an appropriate amount of internal standard to molten phenol and mix thoroughly.

D-3.7.3 Using a preheated chromatographic syringe (approximately 75 °C), inject 1 µl of molten phenol specimen. Phenol is not diluted with water because water may cause ghost peaks.

D-3.7.4 Allow approximately 45 min for all components to elute from the column.

D-3.7.5 When phenol elutes, raise the column oven temperature to 235°C. The column should remain at 235°C for approximately 1 h. Before another chromatograph run is attempted, stabilize the oven at 180 °C for at least 10 min.

D-3.8 Calculation and Report

D-3.8.1 *Calculation*

Determine the concentration of each impurity using the following formula:

$$M_c = (F_c \times A_c \times M_I) / A_I$$

where,

M_c = concentration of impurity C, mg/kg;

F_c = relative response factor for impurity C versus the internal standard;

A_c = area of impurity C;

M_I = concentration of internal standard mg/kg; and

A_I = area of internal standard.

D-3.8.2 Report the concentration of each impurity to the nearest milligram per kilogram.

ANNEX E

(*Clause* 6)

SAMPLING OF PHENOL (CARBOLIC ACID)

E-1 GENERAL REQUIREMENTS OF SAMPLING

E-1.1 In drawing, preparing, storing and handling samples the following precautions shall be observed.

E-1.2 Samples shall not be taken in an exposed place.

E-1.3 The sampling instrument shall be clean and dry when used.

E-1.4 The samples shall be placed in suitable, clean, dry, air-tight, amber or dark blue glass containers or any other container, on which the material has no action.

E-1.5 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, the year of manufacture and other important particulars of the consignment.

E-1.6 Samples shall be protected from light.

E-1.7 Phenolic substances burn the skin and may be absorbed into the system through the skin. The sampler shall wear gloves, preferably of polyvinyl chloride, and also a face shield. Inhalation of the vapours from hot material shall be avoided.

E-2 SCALE OF SAMPLING

E-2.1 Lot

All the containers in a single consignment of material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

E-2.1.1 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately.

E-2.2 Sampling from Containers

The number of containers to be selected for sampling shall be in accordance with Table 4.

E-2.2.1 The containers shall be selected at random. In order to ensure randomness of selection, random number tables shall be used (*see* IS 4905).

E-3 PREPARATION OF TEST SAMPLES

E-3.1 When the material consists of crystallized masses, the containers selected from the consignment shall be allowed to stand in an open tank, on a grid below which a closed steel coil is fitted. When the material is completely melted, stir it thoroughly with a clean, dry, smooth, hardwood agitator, and then draw samples from each by means of dry glass sampling tube from different depths, care being taken to reduce atmospheric exposure to the minimum.

Table 4 Minimum Number of Containers to be Selected for Sampling from Various Sizes of Lots

(*Clause* E-2.2)

Size of Lot	Size of Gross Sample
Under 25	5
25 to 49	5
50 to 99	10
100 to 199	15
200 to 299	20
300 to 499	30
500 to 799	40
800 to 1 299	55
1 300 to 3 199	75
3 200 to 8 000	115

E-3.1.1 Out of the material drawn from each individual container, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample sufficient for carrying out triplicate determinations for all the characteristics specified. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

E-3.1.2 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual sample representing the containers selected shall be for the purchaser, another for the supplier and the third for the referee.

E-3.1.3 All the individual and composite samples shall be transferred to separate sample containers. These containers shall then be sealed air-tight with stoppers and labeled with full identification particulars given in **E-1.5**.

E-3.1.4 The referee test samples, consisting of a composite sample and a set of individual samples, shall bear the seals of both the purchaser and the supplier. They shall be kept at a place agreed to between the two, to be used in case of any dispute.

E-4 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

E-4.1 The purchaser may examine and test each of the individual samples separately for compliance with the requirements of this standard, or he may decide at any stage of the progress of the examination to test a composite sample (*see* **E-3.1.1**) for determining conformity of the lot to this specification.

E-4.2 When the individual samples are separately examined and the results vary from one sample to another, the criteria for judging the quality of the lot for the purpose of acceptance on the basis of the results obtained shall be at the discretion of the purchaser, unless otherwise previously agreed to between the purchaser and the supplier.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc : No. PCD 8 (1051).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 323 01 31, 323 94 02, 323 33 75

Telegrams. Manaksanstha
(Common to
all offices)

Regional Offices:

Telephone

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002
323 76 17
323 38 41

Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola
CALCUTTA 700054
337 84 99, 337 85 61
337 86 26, 337 86 62

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022
60 38 43
60 20 25

Southern : C. I. T. Campus, IV Cross Road, CHENNAI 600113
235 02 16, 235 04 42
235 15 19, 235 23 15

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)
MUMBAI 400093
832 92 95, 832 78 58
832 78 91, 832 78 92

Branches : AHMADABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. PATNA. PUNE. THIRUVANANTHAPURAM.

Printed at New India Printing Press, Khurja, India